The
MERRILL-CROWE PROCESS

SIMULTANEOUS

PRE-COAT CLARIFICATION

DE-AERATION

PRECIPITATION

THE MERRILL COMPANY
Engineers

582 MARKET STREET, SAN FRANCISCO 4, CALIF.

CABLE ADDRESS "LURCO"

MERCO

To Whom It May Concern

The Merrill-Crowe Precipitation
Process and Apparatus used for the
Simultaneous Clarification, De-aera-
tion and Precipitation of Cyanide So-
lutions are protected by Patents and
pending Patent Applications owned
by The Merrill Company in the United
States and in Foreign Countries.

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FOREWORD

In preparing this Catalog we have endeavored to describe the essential features of the Merrill-Crowe Process and apparatus used for the Simultaneous Clarification, De-aeration and Precipitation of cyanide solutions. Limited space has prevented a full description of all of the details involved but we have pointed out the applications of the various types of equipment and the advantages to be expected, and where practical have included the essential operating features.

The Merrill-Crowe Precipitation Process, embodying the Simultaneous steps of Pre-Coat Clarification, De-aeration and Precipitation for the recovery of gold and silver from cyanide solutions is now almost universally used throughout the world. It appears in the flow sheets of all new mills and in addition has been adopted by most of the older plants, effecting substantial savings in labor and reagent cost, and resulting in more efficient precipitation and higher grade bullion. The description which follows indicates how these results are obtained with the several different types of equipment.

Enquiries and requests for quotations should specify the location of property, the nature and tonnage of the material to be cyanided, the tonnage of solution to be precipitated daily, and whether the values to be recovered are principally gold or silver; also whether appreciable amounts of copper are likely to be present in the solution. Where available, a drawing showing the general arrangement of the mill in plan and profile will enable us to suggest the most suitable form of equipment. Charges for Process Rights and Equipment are based upon the above figures, and prices and estimates will be furnished promptly upon receipt of the necessary information.

THE MERRILL COMPANY · SAN FRANCISCO

Mill of the Hudson Bay Mining and Smelting Co. at Flin Flon, Manitoba, where Merrill-Crowe Precipitation and Mills-Crowe Cyanide Recovery are utilized in the cyanidation of a low grade pyritic flotation tailing.
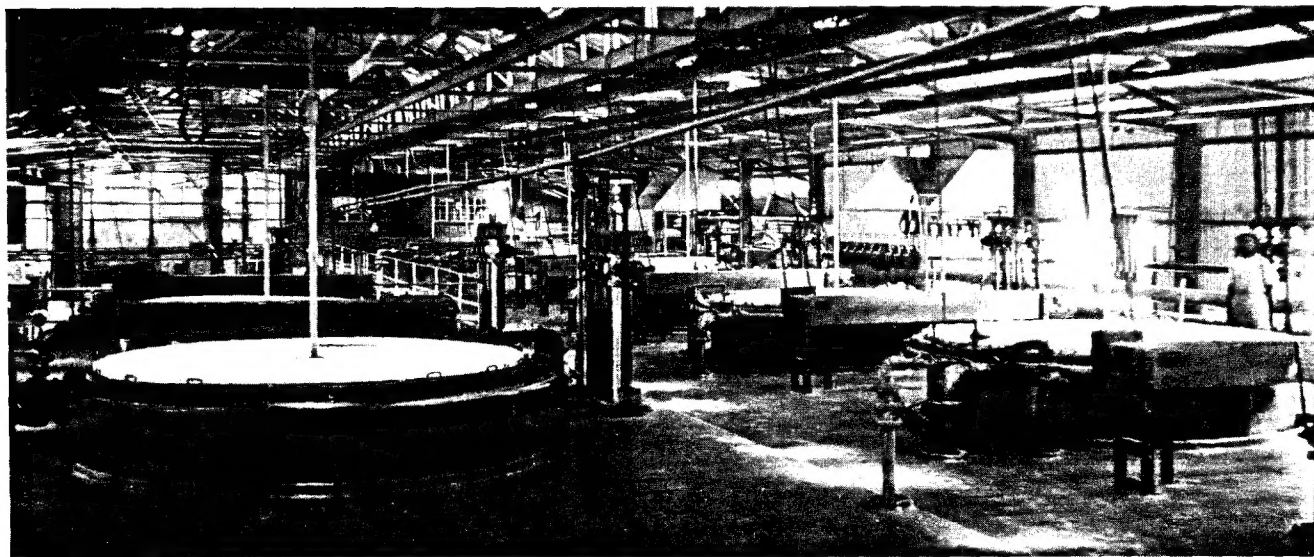
The world's largest precipitation plant. Ten Merrill-Crowe Vacuum Filter units installed at Randfontein, and handling approximately 20,000 tons of solution daily.
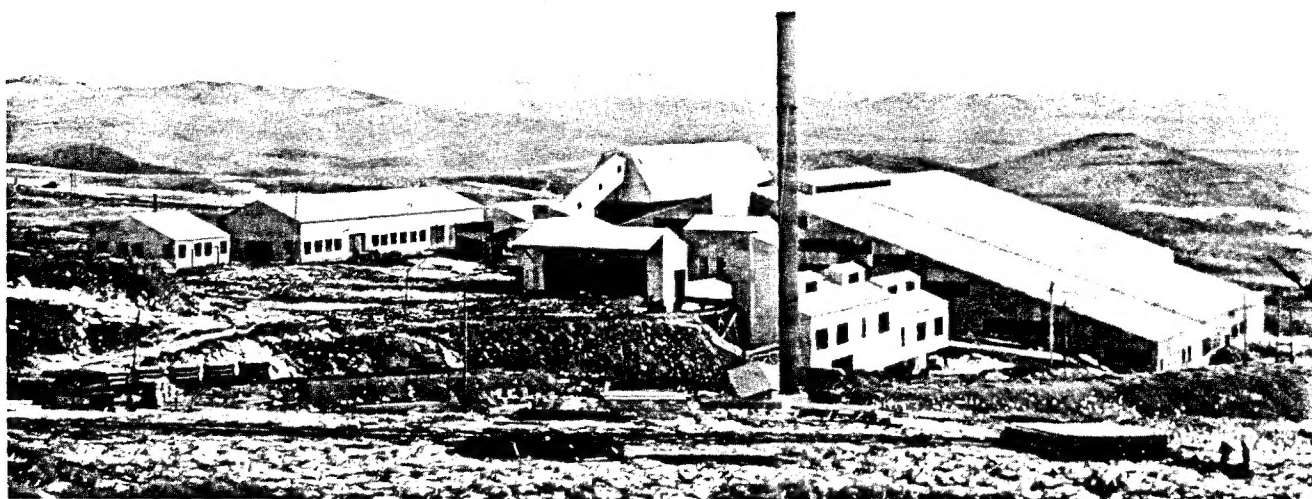
Mill of Getchell Mine, Inc., Golconda, Nevada. This recently completed 600-ton cyanide plant embodies both roasting and cyanidation, with sand leaching and agitation-filtration of slimes. Solutions are clarified and precipitated by the Merrill-Crowe Process.

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MERRILL-CROWE
SIMULTANEOUS CLARIFICATION-PRECIPITATION
PROCESS

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In modern milling processes, cyanidation may be practiced in a variety of ways. Most frequently encountered is the "all slime" plant, grinding in cyanide solution followed by thickening, continuous series agitation and continuous slime filtration, either single or double stage, depending upon ore grade. Of less frequent application is the practice of separating the more sandy portions of the ore for gravity leaching as in the older plants on the Rand, in the Mysore field in India, and at the Homestake South Dakota, where the necessity for thorough air oxidation of certain constituents of the ore render this treatment particularly effective. If amenable to flotation, the ore may be ground in water and floated, with subsequent cyanidation of the flotation concentrate only, as at McIntyre and Pamour in Ontario, and in Australia where the concentrates are roasted prior to cyaniding. However, not many gold ores will yield a satisfactory tailing by flotation alone and consequently both tailing and concentrates are separately cyanided. In all of the above flow sheets, where the ore contains appreciable amounts of free gold, various devices are used to recover as much as possible of the metal in the grinding circuit; hence the use of blanket strakes, riffles, traps or jigs in many gold mills.

However, for precipitating or recovering the metallic values from cyanide solutions, no method has yet been found to compete successfully with the Merrill-Crowe Process in which the metals are precipitated on zinc dust immediately following Simultaneous Pre-Coat Clarification and De-aeration, with substantially complete removal of suspended solids and colloidal precipitates, and of dissolved oxygen.

In Africa and India a few of the older mills still use zinc shavings for the precipitation of gold but in Africa particularly, these plants are gradually changing over to the more effective Merrill-Crowe Process.

Sodium sulphide precipitation of silver at Cobalt, Ontario, and charcoal precipitation of gold at Youanmi, Australia, were both used successfully for a time but did not prove applicable elsewhere. Precipitation of silver on aluminum dust was also practiced at Cobalt but while attempted at several other large plants in Mexico and South America, was discontinued owing to difficulties in filtering and melting, caused by the formation of calcium aluminate in the solutions. Electrical precipitation has been repeatedly tried but power and operating costs are prohibitive and a "barren" tail solution cannot be obtained.

The efficient precipitation of cyanide solutions with zinc dust is dependent upon two essential preliminary or conditioning steps, namely, thorough clarification and the removal of dissolved oxygen. By thorough clarification is

meant not merely removal of suspended ore slimes, such as are plainly visible to the naked eye, but also the more harmful colloidal hydrates of aluminum, iron and magnesium, which form and precipitate out in most pregnant solutions, particularly on standing for any appreciable length of time. To remove such deleterious solids the Merrill-Crowe Process uses submerged vacuum leaf filters of improved design and equipment for Pre-Coating the filter surfaces with diatomaceous silica. The latter is a comparatively recent development and is described in more detail on page 9. The outlet of the clarifying filter is connected directly to the Crowe De-aerating tower, so that immediately after thorough clarification and partial de-aeration, the solution enters the main vacuum chamber, where the removal of oxygen is completed.

The chemical reaction involved when gold is precipitated by metallic zinc is essentially one of reduction by nascent hydrogen which is formed by the action of alkali cyanide upon the zinc. Most cyanide solutions resulting from the treatment of ores contain appreciable amounts of dissolved oxygen and if this oxygen were allowed to remain, it would immediately combine with the evolved hydrogen, increasing the consumption of zinc and cyanide with the formation of calcium zincate and other insoluble compounds of zinc and cyanide which seriously interfere with precipitation. The whole operation of precipitation and subsequent recovery of the precipitated metals is simplified and rendered more efficient by the simple expedient of mechanically removing this interfering element. In the Merrill-Crowe Process this is accomplished by passing the solution through suitable towers in which the solution is exposed in thin films to the action of a high vacuum. The decreased solubility of oxygen at low absolute pressure causes rapid evaporation of the oxygen, and cyanide solutions thus treated will usually contain no more than traces of oxygen.

Immediately following Clarification and the removal of free oxygen, the solution is contacted with finely divided metallic zinc, under conditions which preclude the re-absorption of oxygen. This results in maximum efficiency of the precipitant, since no hydrogen is wastefully consumed in combining with dissolved oxygen. The mixture

Merrill - Crowe Precipitation Unit at the tailings plant of the Kennedy Mining and Milling Co., Jackson, California.

Simultaneous Clarification, De-aeration and Precipitation equipment, with Bag Type Filters, handles the solution resulting from the treatment of approximately 1000 tons of combined sand and slime tailings daily.
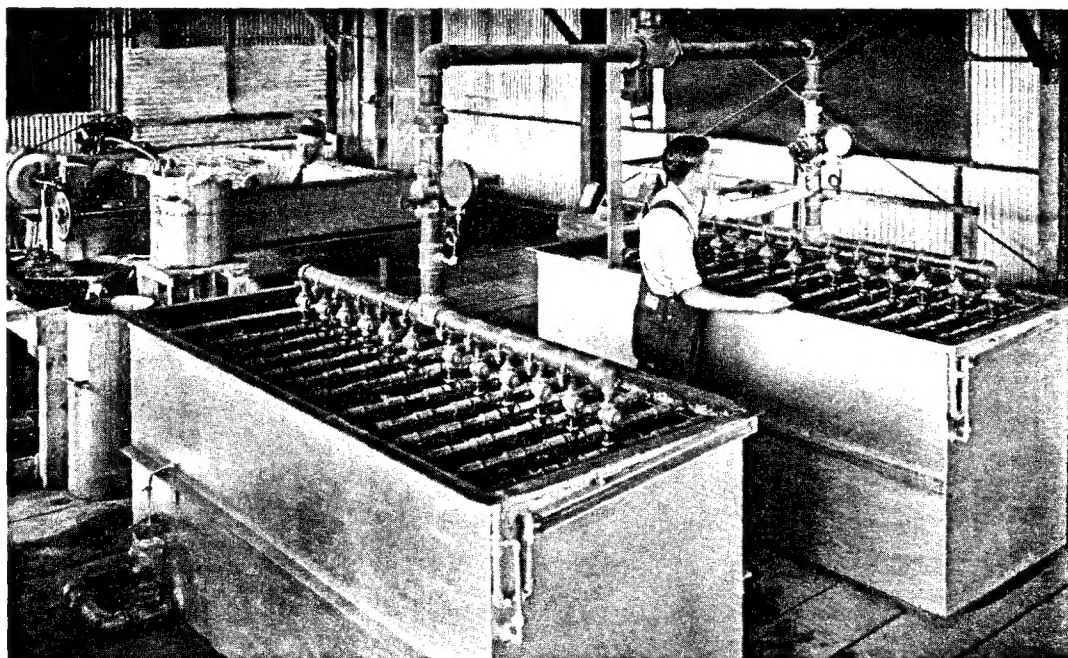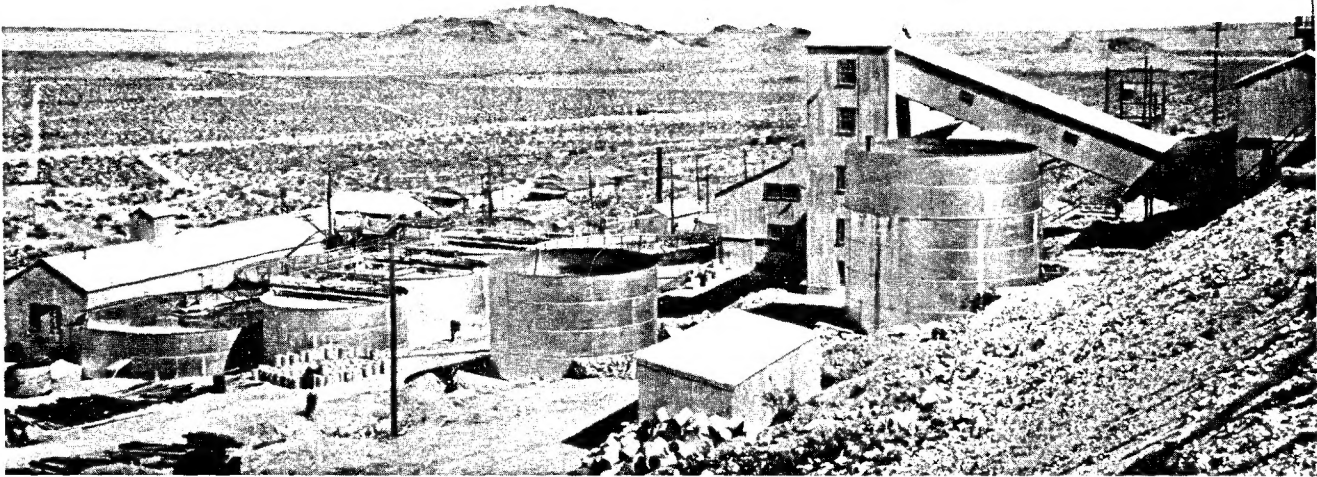
Golden Queen Mill, Mojave, California. This thoroughly modern all cyanidation plant has a rated capacity of from 350 to 400 tons of ore per day, and the equipment includes a Merrill-Crowe Simultaneous Clarification-Precipitation Unit with a daily capacity of 1000 tons of solution.

of precipitated metals and zinc dust is collected on a suitable filter, so designed that a uniform layer of precipitant is deposited over the whole filter surface, whereby each particle of solution is brought into intimate and equal contact with the precipitant. The accumulated precipitate of gold and silver is periodically removed for melting and refining and the precipitated or barren solution is returned for re-use in the milling process.

As distinguished from zinc shavings precipitation, a complete recovery of the precipitated values is made at each clean-up. With gold solutions, the values returned to the boxes with the washed shavings usually approximate one-half of the gold cleaned up; with silver the absorption in the boxes is less but in neither case can it be prevented without excessive consumption of zinc and chemicals. The loss in interest on values thus continually held back is a direct charge against the old shavings process. All operators will, of course, appreciate the advantages of the complete clean-up of the Merrill-Crowe Process as a means of checking bullion production against ore and solution assays. Frequent and complete clean-ups are of course particularly desirable in mills which handle custom ore. The compactness and flexibility of the plant effects a marked saving in floor space and building construction and in addition, in the colder countries such as Northern Canada, a considerable economy in heating during the winter months.

The Merrill-Crowe Precipitation Process is now almost universally used in Cyanide plants throughout the world. Whether for the treatment of silver ores in Mexico, for gold ores in the rapidly developing districts of Canada, or on the Rand, the most important gold mining area of the world, the Process will be found in operation. In the latter district, the total amount of solution precipitated by the Merrill-Crowe Process, approximates 200,000 tons per day. One mill, Randfontein, accounts for 20,000 tons of this solution. In many of the older plants, zinc shavings boxes have been dismantled to be replaced with the Merrill-Crowe Process, and in designing new plants, metallurgists on the Rand, and in fact throughout the world, have, almost without exception, embodied it in their flow sheets.

Merrill-Crowe Precipitation Plant
FILTER PRESS TYPE
DRAWING No. 1
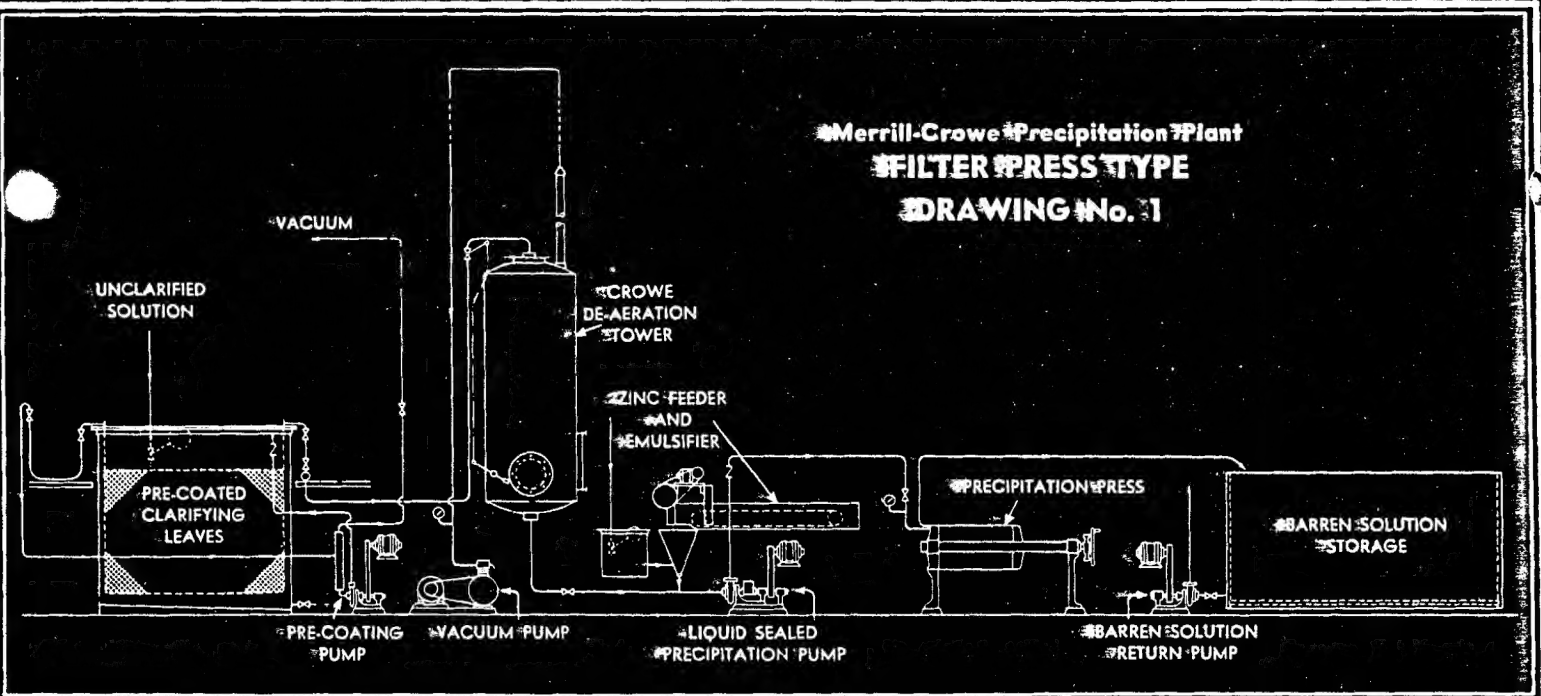
MERRILL-CROWE Simultaneous Pre-Coat Clarification-Precipitation

TYPES OF EQUIPMENT USED

To users of the Merrill-Crowe Process we offer three different types of equipment, the choice of which is dependent upon the size of the plant, that is, upon the tonnage of solution handled daily, upon the nature and quantity of the metals to be precipitated, and, lastly, upon the preference of the individual operator. General arrangement of the milling plant may also be a determining factor. There is some variation in first cost of the three types but all are substantially equal in operating costs and in metallurgical efficiency.

In each type, the equipment used for Simultaneous Pre-Coat Clarification and De-aeration is identical as will be noted in the drawings and descriptions which follow. It is only in the filters used for collecting the precipitate that the three types differ. Each type takes its name from the precipitate filter used, that is, "Filter Press Type," "Bag Filter Type" and "Vacuum Leaf Filter Type."

The Filter Press Type has the most general application and with the recently developed improvements in Clarification, is preferred by most operators. Except for small plants the cost of the Press Type is not appreciably greater than the Bag Type and is considerably less than for the Vacuum Leaf Type, which is used principally in large plants or under exceptional conditions where the larger filter area provided is particularly advantageous.

FILTER PRESS TYPE

The equipment used for the Simultaneous Clarification, De-aeration and Precipitation of cyanide solutions, using Pre-Coated clarifying leaves, and filter presses for collecting the precipitate, is shown in Drawings No. 1 and No. 2, which represent two of the arrangements most frequently used.

In each case, a single, liquid-sealed centrifugal pump is used to effect the passage of the solution through the successive steps of clarification, de-aeration and precipitation. The clarifying tank is kept filled to a constant level with unclarified gold-bearing solution, the inflow to the tank being controlled by an automatic float valve as in Drawing No. 1. Alternatively, a shallower tank may be used with provision for constant overflow. This overflow may be surplus primary thickener overflow going to mill storage, or solution from the pregnant storage tank pumped to the clarifier with surplus overflow returned to storage. Suspended in the clarifying tank are the vacuum clarifying leaves with outlets connected to a manifold which in turn is connected to the top of the vertical Crowe vacuum tower in which final de-aeration of the solution is effected.

Rectangular steel clarifying tanks have now come into quite general use and these tanks are built with a separate compartment in which the individual filter elements are pre-coated with a thin layer of diatomaceous silica as a filter aid. The filter leaf, after washing, is immersed in the pre-coating compartment, to which has been added a small amount of the pre-coating material, kept in agitation with compressed air. By means of a flexible hose, the leaf is attached to the suction side of a small self priming centrifugal pump, which withdraws solution through the leaf, depositing the filter aid on the filter fabric, and returning the effluent to the tank. After pre-coating, the leaf is lifted, returned to its operating position in the clarifying tank, and again connected to the discharge manifold. Throughout the transfer and re-immersion continuous vacuum is maintained on the leaf so that the layer of filter aid is not detached or disturbed, and after re-immersion, circulation through the leaf is continued until the effluent is perfectly clear, when the valve leading to the manifold is opened and regular operation resumed. Thus any imperfection in clarification is readily detected and solution is not passed on to the subsequent steps of de-aeration and precipitation until perfect clarity is assured.

Inflow of solution to the Crowe tower and consequently the solution level in the tower, is controlled by an automatic float valve. Within the tower the solution passes down over suitable grids which break up the flow into small streams and films, thus effecting the substantially complete removal of dissolved oxygen. The top of the de-aerating tower is connected with a dry vacuum pump which maintains a high vacuum within the tower and removes the air released from the solution. Clarified, de-aerated solution is withdrawn from the bottom of the tower by a single stage centrifugal pump, liquid-sealed to prevent re-entry of air through the pump gland.

A belt type zinc feeder with integral motor drive discharges a regulated amount of zinc dust into a mixing cone supplied with suitable solution, preferably barren solution from a small float controlled steady head tank. A liquid reagent feeder supplies a continuous and uniform feed of soluble load salt, preferably lead nitrate, for activating the zinc. This feeder may be positioned to discharge into a steady head tank or to the clarifying tank, the choice being governed by the nature of the solution to be precipitated.
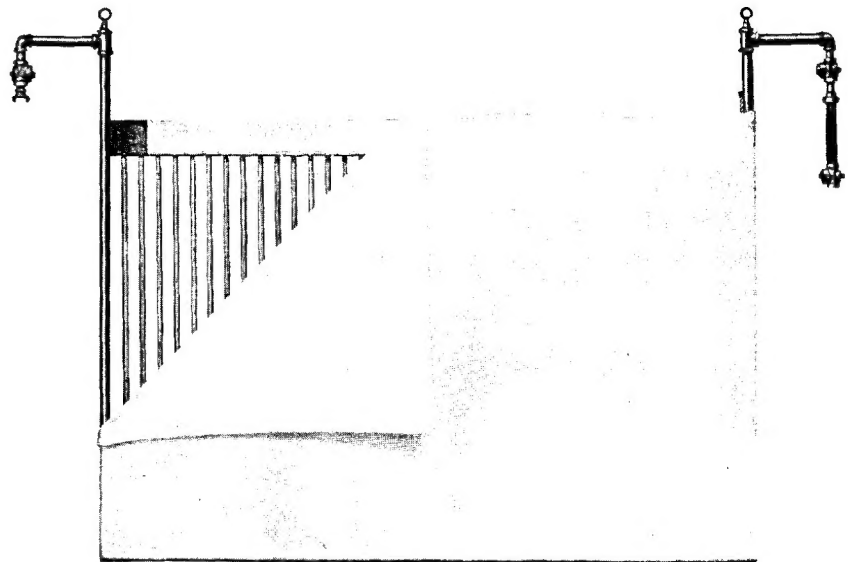
Drawing No. 2 shows the activated zinc emulsion being forced into the main solution line entering the press by means of a small triplex pump. Alternatively, as in Drawing No. 1, the outlet of the emulsion cone is controlled by a mechanically operated valve by which zinc emulsion, at regular intervals, is introduced directly into the suction of the main solution pump and thence into the precipitation press.

The filter presses now used in the Merrill-Crowe Process are of conventional design with square containers and with closed discharge, which not only avoids splash and frequent discharge cock renewals but also makes it possible to elevate the barren solution several feet above the level of the press and so discharge directly into a barren solution storage tank placed on the same level.
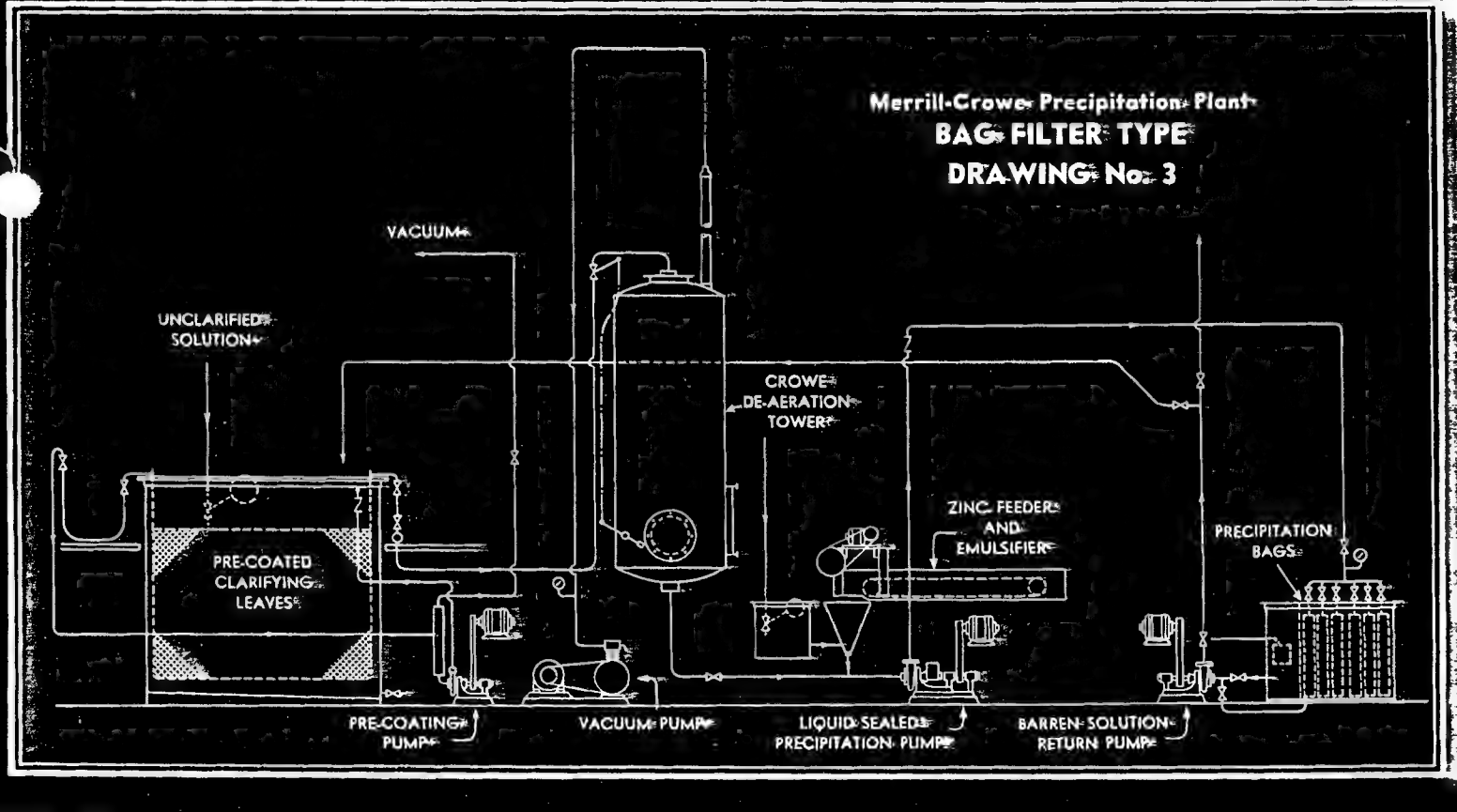
Merrill-Crowe Precipitation Plant
FILTER PRESS TYPE
DRAWING No. 2
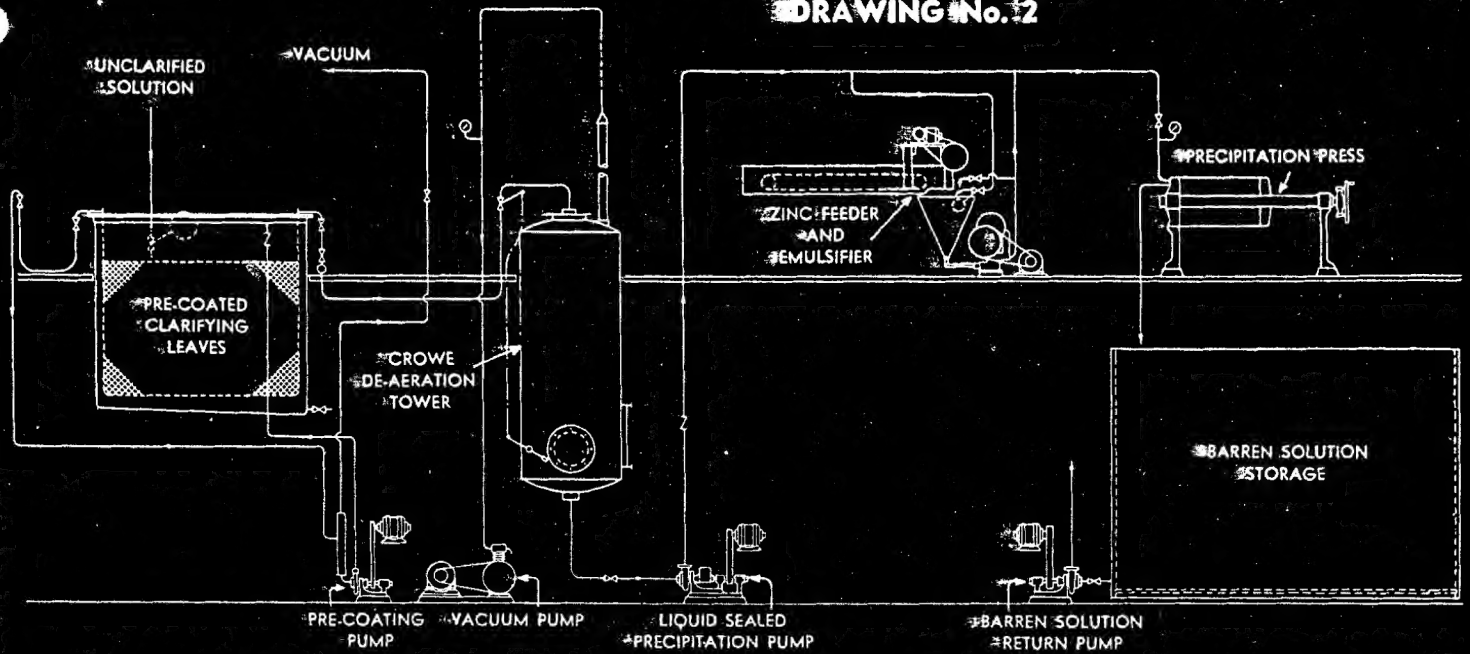
Photograph showing construction of the new Merrill-Crowe Clarifying Leaf. Circular wood splines are firmly mounted in the wood header and lower perforated drainage pipe. These filters are usually operated with sheeting or light canvas cover only but an unstitched burlap under cover is sometimes used.
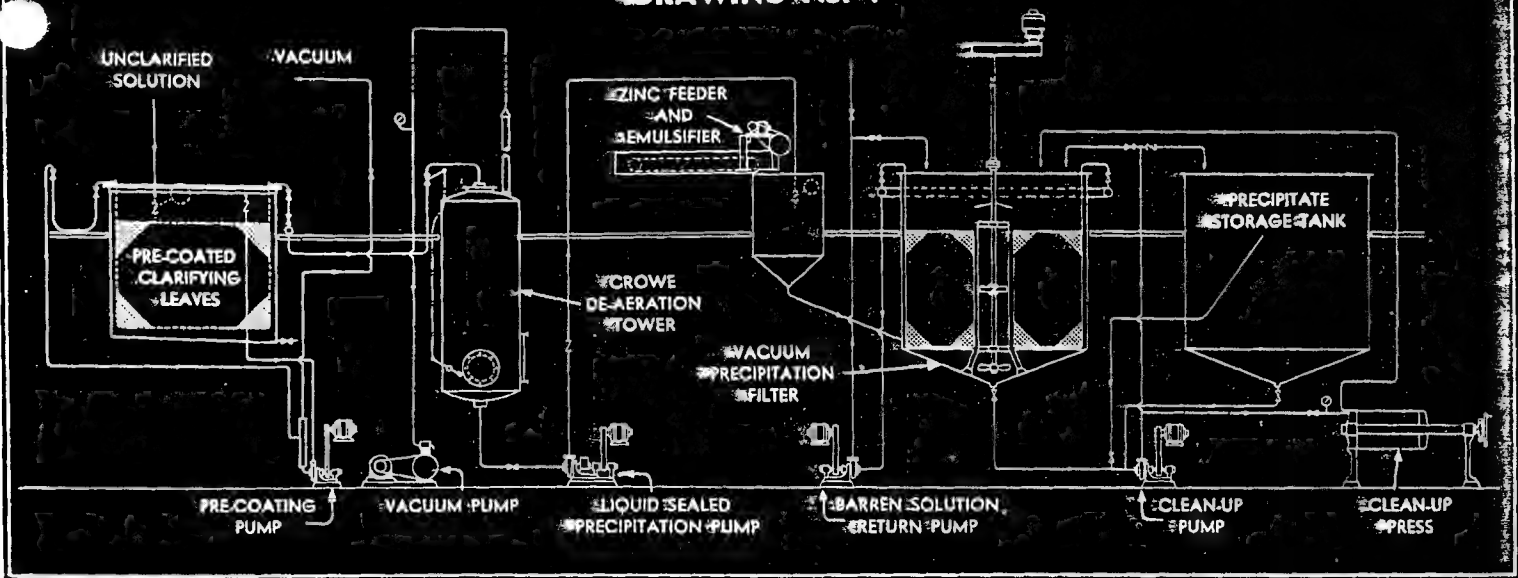

VACUUM FILTER LEAF

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Labels in drawing:
VACUUM
UNCLARIFIED SOLUTION
CROWE DE-AERATION TOWER
ZINC FEEDER AND EMULSIFIER
PRECIPITATION BAGS
PRE-COATED CLARIFYING LEAVES
PRE-COATING PUMP
VACUUM PUMP
LIQUID SEALED PRECIPITATION PUMP
BARREN SOLUTION RETURN PUMP

BAG FILTER TYPE

Drawing No. 3 shows the usual arrangement of equipment in a Merrill-Crowe Plant with Pre-Coat Simultaneous Clarification-Precipitation using Bag Type precipitate filters. These filters are particularly suitable where small solution tonnages are handled. With the recently developed Fitzgerald Bag Head the bag units are quickly assembled and with the duplicate set of heads provided, clean-up is effected with minimum loss of time. The bags are contained in a steel tank, provided with a locked cover to prevent tampering or theft, and a centrifugal pump with automatic float control elevates the precipitated solution to the barren solution storage. Alternatively, the barren solution may gravitate to the storage tank. Clean-up is made by emptying the tank, draining and blowing the bags with compressed air, and then disconnecting the bags and removing the inner filters containing the precipitate, which is dried, fluxed and melted in the usual way.

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BAG FILTER UNIT

Merrill-Crowe Precipitation Plant
VACUUM LEAF TYPE
DRAWING No. 4

UNCLARIFIED SOLUTION • VACUUM • ZINC FEEDER AND EMULSIFIER • PRECIPITATE STORAGE TANK • PRE-COATED CLARIFYING LEAVES • CROWE DE-AERATION TOWER • VACUUM PRECIPITATION FILTER

PRE-COATING PUMP • VACUUM PUMP • LIQUID SEALED PRECIPITATION PUMP • BARREN SOLUTION RETURN PUMP • CLEAN-UP PUMP • CLEAN-UP PRESS

General arrangement of a Vacuum Leaf Type plant is shown in Drawing No. 4. Clarified, de-aerated solution, withdrawn from the Crowe receiver by the liquid-sealed centrifugal pump, discharges into a steady head tank, to which is also added zinc from the precipitant feeder. The mixture of solution and precipitant enters the precipitation tank and is continuously circulated over the submerged vacuum filter leaves, the barren solution being withdrawn through the leaves by a centrifugal pump which in turn discharges to a suitable barren solution storage tank. Access to the precipitation tank is only required at a clean-up and, therefore, the tank is provided with a locked cover of heavy screen to prevent theft. When clean-up is made, the precipitation tank is emptied and the precipitate washed from the leaves and transferred by pump to a settling tank. The bulk of the solution is decanted and returned to the precipitation circuit after which the settled precipitate is transferred to a small filter press for drying. Owing to the constant circulation maintained in a precipitate filter of this type, the filter surfaces are coated with a very uniform layer of precipitant which of course results in the highest possible precipitating efficiency. Without exception this is the equipment used in all of the recently built cyanide plants on the Rand, and many similar installations have been made in other parts of the world. It is particularly suitable for large plants, especially where, as on the Rand, the gold precipitate is acid treated; also where a considerable bulk of silver precipitate is to be handled.

CROWE
DE-AERATING TOWER

(12)
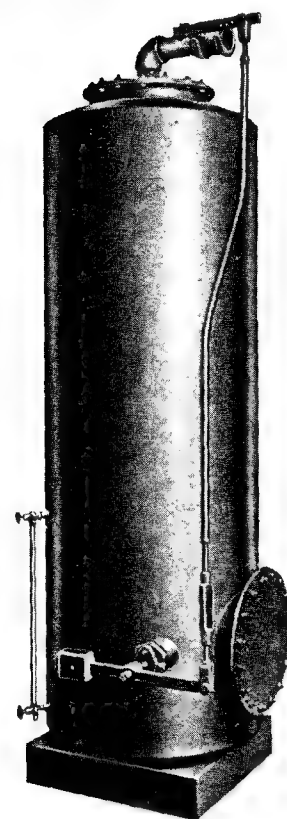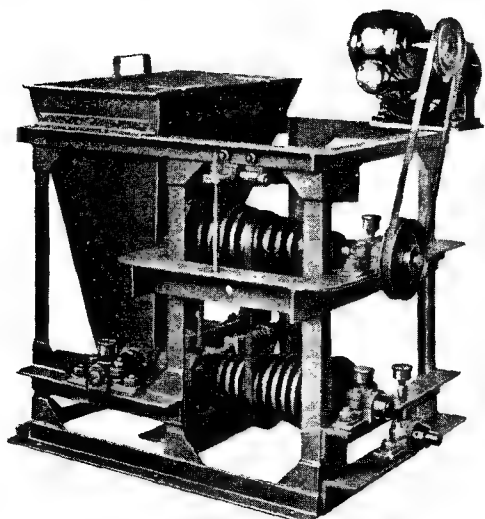
SPIRAL TYPE ZINC FEEDER
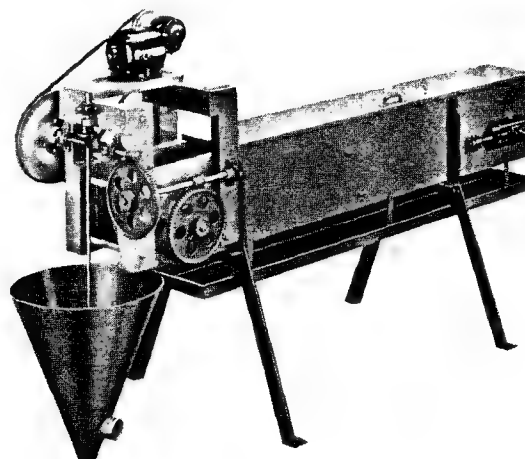
BELT TYPE ZINC FEEDER

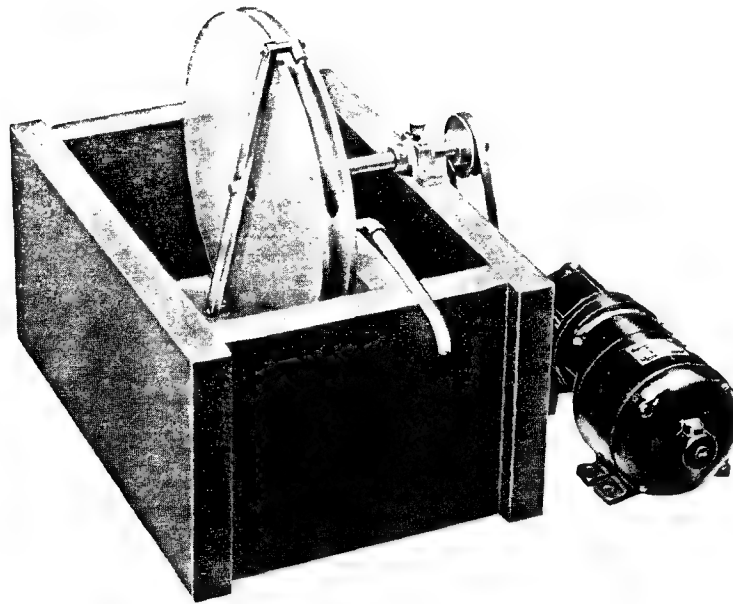# "MERRILLITE" Zinc Dust Precipitant

With the high efficiency now developed by the Merrill-Crowe Precipitation Process, resulting in extremely small quantities of zinc used to precipitate the metals from solutions, it has become increasingly important to use a zinc dust of high metallic content and uniformity. Also the mesh of zinc dust should be as fine as practicable in order to obtain the maximum surface contact between the zinc and the solution. We know of no zinc dust that fulfills these requirements as satisfactorily as that manufactured by The Alloys Company, a subsidiary of the Merrill Company. Constant research is maintained in an endeavor to supply precipitants best suited for use with the Merrill-Crowe Process.

THE FAMILIAR
MERRILLITE CONTAINER

This zinc dust is produced in San Francisco and is marketed under the trade-mark "Merrillite." Many of the principal cyanide plants of Canada, the United States, Mexico and other parts of the world use this zinc dust exclusively with entirely satisfactory results, and we have no hesitancy in giving our unqualified recommendation to this product for use with the Merrill-Crowe Process. In particular we advise its use in the initial operation of all new plants, because we know that MERRILLITE, being of well established uniformity and unquestioned high efficiency, will give dependable precipitation, thus avoiding the possibility of variations which may result from the use of other brands of zinc dust.

LIQUID REAGENT FEEDER

Above is shown the New Liquid Reagent Feeder used in the Merrill-Crowe Process for feeding lead nitrate or acetate. Also to supply a uniform strong cyanide drip when needed. Built of redwood and stainless steel with nothing to rust or wear out and supplied with or without reduction motor drive.

Below are shown two sizes of the Sealed Pumps used to withdraw de-aerated solution from the Crowe Vacuum Receiver. These are supplied in several different types and in various sizes dependent upon the tonnage of solution to be handled.
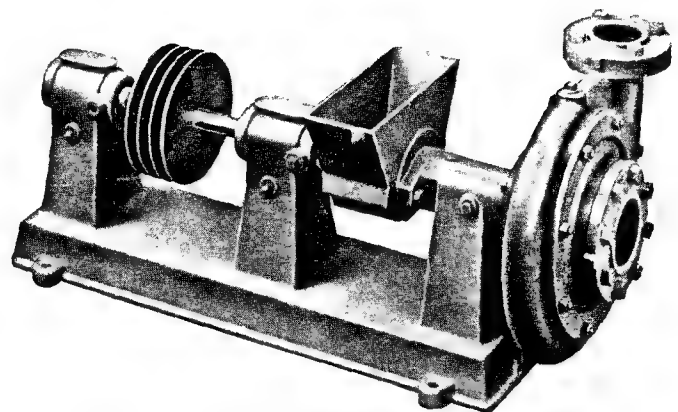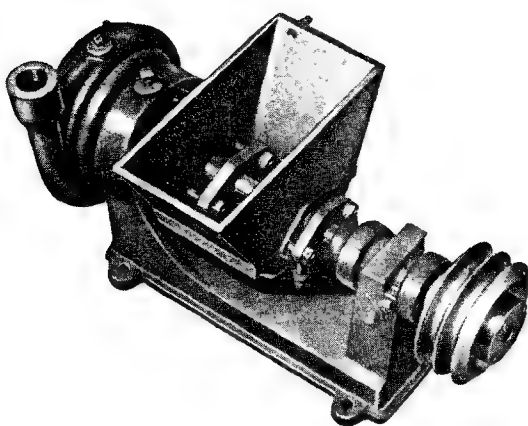
LIQUID SEALED CENTRIFUGAL PUMPS

MERRILL-CROWE
PRECIPITATION PROCESS

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Details of Operation

Efficient precipitation of gold and silver is dependent upon the proper control of certain physical and chemical properties of the solution. The most important of these properties are listed below.

SUSPENDED SOLIDS, such as ore slime and precipitates of calcium carbonate, with hydrates of aluminum, magnesium and iron, present in the pregnant solution before clarification. These combined solids should be completely removed by efficient clarification.

SUSPENDED PRECIPITATES, as above defined, which may continue to form in the solution AFTER clarification. This formation occurs slowly and is almost completely prevented by clarifying and pre-c   ating simultaneously.

SCALE FORMING COMPOUNDS, chiefly the carbonates and sulphates of lime.

OXYGEN AND CARBON DIOXIDE GASES DISSOLVED IN THE SOLUTION.

CYANIDE STRENGTH AND ALKALINITY.

CLARIFICATION

Perfect clarification is probably the most important single factor in obtaining efficient precipitation. It should remove all suspended solids from the pregnant solution, even those colloidal suspensions invisible to the naked eye. Long experience has shown that such clarification is best effected in low pressure vacuum leaf filters of special design to facilitate handling and changing covers. Details of this leaf are shown on page 10, and a drawing and description of the complete unit appears on pages 8 and 9. This includes the equipment used in the recently developed Pre-Coat conditioning of the clarifier leaves, without which the present perfection of clarification would not be possible.

Since filtration is effected under a negative pressure, partial removal of dissolved oxygen has already been effected when the solution enters the Crowe receiver, where the deoxygenation is completed. By thus clarifying and de-aerating simultaneously, the capacity of the vacuum receiver is considerably increased, and the substantially complete removal of oxygen is greatly facilitated.

Pre-Coat Clarification plays another important role in preparing solution for precipitation. Carbonates and sulphates of lime are present in varying amounts in all cyanide solutions and tend to deposit scale on all exposed surfaces. Conditions within the precipitate filter cake are particularly favorable for such deposition and the deposit thus formed not only covers the zinc surfaces and increases the consumption but also impedes the flow of solution through the filter fabric, causing high pressure and necessitating frequent removal of the filter cloth.

With the advent of Pre-Coat Clarification this difficulty is minimized because a large proportion of the scale forming compounds, which might cause later trouble in the precipitate filter, are deposited on the enormous surface areas of the diatomaceous silica used as a Pre-Coat layer on the clarifying leaves. Since these filters are frequently washed and the layer of Pre-Coat renewed, the deposition and elimination of the scale at this point is highly desirable.

In plant operation one or more of the clarifying leaves should be removed daily and the adhering layer of Pre-Coat and slime washed off with a jet of water or solution. The Pro-Coat layer greatly facilitates this washing, besides prolonging the life of the filter fabric and maintaining its permeability, i. e.: the filtering capacity of the leaf.

In starting new plants, all solution tanks and pipe lines should be thoroughly washed out with lime water to remove oil and grease which otherwise will cause serious pressure trouble in the clarifying and precipitating filters.

THE OPERATOR SHOULD KEEP IN MIND THAT IMPERFECT CLARIFICATION IS THE MOST FREQUENT CAUSE OF PRECIPITATION TROUBLE AND THAT CAREFUL ATTENTION TO CLARIFICATION DETAILS WILL BE WELL REPAID IN IMPROVED PRECIPITATION RESULTS. THE CLARIFIED SOLUTION MUST BE NOT MERELY "CLEAR" BUT "BRILLIANT" LIKE THE BARREN SOLUTION ISSUING FROM THE PRECIPITATE FILTERS, AND NO WASHED AND PRECOATED LEAF SHOULD BE RECONNECTED TO THE MAIN VACUUM MANIFOLD UNTIL THE CIRCULATING EFFLUENT SHOWS SUCH CLARITY.

DE-AERATION or REMOVAL OF DISSOLVED GASES

Efficient and complete precipitation of metals from cyanide solutions requires that the solution, after clarifying, be further conditioned by the substantially complete removal of dissolved oxygen. Complete precipitation cannot take place in the presence of even traces of free oxygen. Hydrogen, formed when metallic zinc precipitant dissolves in alkaline cyanide solution, will combine with and nullify the effect of free oxygen, but at the expense of a wasteful consumption of zinc and cyanide, and the formation of troublesome by-products. In the Merrill-Crowe Process, the oxygen is mechanically removed in a preliminary conditioning step, by subjecting the solution to the action of a high vacuum. Carbon dioxide is simultaneously removed, thus minimizing the formation of calcium carbonate in the precipitate filter.

SIMULTANEOUS CLARIFICATION, DE-AERATION and PRECIPITATION

The remarkable efficiency of the Merrill-Crowe Precipitation Process is largely due to Simultaneous Clarification and De-aeration of the solution, followed by the immediate addition of suitably activated zinc dust and precipitation of the metals without rest and without exposing the solution to re-absorption of atmospheric oxygen. Most cyanide solutions after clarification, will, upon standing even for a short time, throw out suspended colloids, consisting largely of the hydrates of alumina, magnesia, and iron. Although hardly visible to the naked eye, these precipitates frequently form in sufficient quantity to coat and insulate the zinc, increasing the pressure in the precipitate filters and seriously interfering with precipitation. Under certain conditions a colloidal precipitate of copper cyanide (Cu_2Cy_2) is similarly formed. These difficulties are minimized and in most cases entirely prevented by Simultaneous Clarification, De-aeration and Precipitation.

CYANIDE STRENGTH and ALKALINITY

Alkaline cyanide solutions resulting from the treatment of most ores will usually contain appreciable amounts of free cyanide and lime. Generally speaking, the cyanide content required for maximum dissolution of gold and silver, is greater than that required for effective precipitation, which on a previously de-aerated solution need be no more than sufficient to dissolve the requisite amount of zinc to cause precipitation and to hold in solution the by-product compounds, such as zinc hydrate, which are

DETAILS OF OPERATION—Continued

formed when metallic zinc is dissolved in cyanide solution.

The "Banket" ore of the Rand is a notable exception to the above statement. When this ore is finely ground, the gold dissolves rapidly in very weak cyanide, so weak, in fact, that it is customary to add a small amount of fresh cyanide to the solutions before precipitation. If otherwise properly conditioned, it may be stated that solutions titrating 0.1 lb. or more NaCy per ton are readily precipitated, and that even as little as 0.05 lb. NaCy may frequently be sufficient. However, where these minimum strengths are used, it will be found that a continuous drip of strong cyanide solution added to the zinc emulsifier will sometimes be helpful in activating the zinc and ensuring trace barrens. The lower cyanide strengths are more favorable to the precipitation of copper and where substantial amounts of this metal are dissolved from the ore, the pregnant solution should contain sufficient free cyanide to hold the copper in solution.

The chief functions of lime used in cyanidation are to neutralize acid cyanicides in the ore and to promote coagulation and settlement of the ore pulp to facilitate filtration and to assist clarification of the decanted solution. Less frequently, high lime content may be required to decompose the sulphides of silver or the sulphotellurides of gold. So far as precipitation is concerned, very little lime is needed; merely enough to ensure the solubility of the added lead salt as alkali plumbite and to coagulate the zinc precipitate to ensure rapid and perfect filtration. Unnecessarily high lime during precipitation is apt to cause a wasteful consumption and hydration of the zinc, according to the following equation:

$$Ca(OH)_2 + Zn = CaZnO_2 + 2H$$

The calcium zincate thus formed is soluble if sufficient cyanide is present, but otherwise remains in suspension or as a coating on the zinc surfaces, thus seriously interfering with precipitation. This and imperfect clarification are the principal causes of excess pressure in precipitate filters. In treating silver ores, the solutions must of course contain sufficient alkali to decompose the double cyanide formed when silver is precipitated on zinc, and to re-form alkali cyanide after precipitation. As pointed out elsewhere, it may be desirable to raise or lower the alkalinity, for the purpose of controlling deleterious colloids or precipitates occurring in some solutions. Any such change should be made GRADUALLY by altering the lime feed to the grinding circuit or to the agitators; do not add lime to pregnant solution storage tank or to clarifier. Proper alkalinity for any given ore or solution can only be determined by actual trial and experience.

FEEDING ZINC DUST

Equipment for feeding zinc dust is now well standardized and is shown in the photographs on page 13. The spiral feeder is sometimes used but the belt type is usually preferred. Either clarified and de-aerated pregnant or barren solution may be used to form the zinc emulsion, the amount approximating one to two g.p.m. for small plants and 3 to 5 g.p.m. for larger tonnages.

The amount of zinc dust required varies with the character of the solution, with the nature and amount of impurities present and the metals to be precipitated. On clean gold ores the zinc dust consumption varies from .02 lb. to .06 lb. per ton of solution. These figures will be increased by the presence of appreciable amounts of silver or copper. In the cyanidation of silver ores, where the solutions may contain several ounces of silver per ton, the zinc consumption will approximate 0.6 oz. per ounce of silver precipitated.

USE OF LEAD SALTS

The precipitation of nearly all gold solutions and of some silver solutions is improved by the addition of a soluble lead salt, which immediately precipitates metallic lead on

DETAILS OF OPERATION—Continued

the zinc, forming a very active zinc-lead couple and stimulating precipitation of the precious metals. Either lead nitrate or lead acetate may be used although the former is preferable since it is more readily soluble. With some cyanide solutions, the addition of a soluble lead salt, either nitrate or acetate, has a tendency to form insoluble lead compounds. The character of this precipitate varies in different solutions and may contain lead cyanide, sulphate, chloride, or basic lead carbonate. These precipitates tend to choke the precipitate filters and may seriously interfere with precipitation. Therefore where such conditions exist, the lead salt should be added to the solution before clarification, either to the pregnant solution storage tank or to the inlet of the clarifier, where the precipitated lead compounds are filtered out. Where no appreciable amount of insoluble lead precipitate is formed, the lead salt may be added in the form of a continuous drip to the zinc emulsifier, where it is immediately precipitated as a black film upon the zinc particles. As pointed out elsewhere, a simple form of liquid reagent feeder is used to add the lead solution either to the zinc emulsifier, to the clarifier, or to the pregnant storage tank as desired.

The amount of lead salt used varies widely, depending upon the nature of the solution being precipitated. On gold solutions the quantity may vary from as little as 5% to as much as 25% of the weight of zinc used. In treating ores containing lead which is soluble in alkaline cyanide solution, as for example silver ores containing lead carbonate, no additional lead salt is required. Similarly, where the solutions contain appreciable amounts of silver or copper, the added lead salt may not be needed, since these metals themselves form efficient precipitating couples with the zinc, and a porous, spongy precipitate which filters readily.

The successful use of lead salts requires careful supervision at all times, since if added at the wrong place, or not

uniformly coated upon the zinc, more harm than good may result. We have already stated that imperfect clarification is the most frequent cause of precipitation trouble; improper use of lead salts is probably the next. Careful study of both will well repay the operator.

EFFECT OF COPPER ON PRECIPITATION

The presence of appreciable amounts of copper in cyanide solutions is usually accompanied by high cyanide consumption and may also decrease extraction if allowed to accumulate beyond certain limits. It seriously interferes with zinc shavings precipitation, causing plating or coating of the zinc and stopping precipitation of the precious metals. However, in the Merrill-Crowe Process, using zinc dust precipitant, the presence of copper has no detrimental effect other than to increase the zinc consumption and lower the grade of precipitate produced. Because new, fresh surfaces are exposed by the constant addition of finely divided zinc, precipitation of the gold and silver is complete, irrespective of the copper present.

EFFECT OF LEAD ON PRECIPITATION

As already stated, the presence of small amounts of lead in solution is beneficial to precipitation. However, in cyaniding ores containing appreciable amounts of soluble lead such as lead carbonate, the amount of lead dissolved and precipitated may cause excessive consumption of zinc and a low grade or base precipitate which is difficult to refine. Lead does not readily combine with alkaline cyanide; the amount dissolved is determined by the lime present to form calcium plumbite. By keeping the lime at the minimum required for settlement, dissolution of lead can usually be kept down to a reasonable amount.

TREATMENT OF PRECIPITATE

The character and grade of precipitate produced in the Merrill-Crowe Process will depend upon several factors.

The most important is the nature of the ore being treated — the amount of gold and silver and of base metals, copper and lead dissolved by the solutions. Clarification, of course, determines the amount of impurity such as ore slime; with the advent of Pre-Coat Simultaneous Clarification Precipitation this is now almost negligible. De-aeration and chemical control of the solution will largely determine the amount of zinc and zinc by-products in the precipitate. With clean gold ores the product may contain from 60% to 90% gold and not to exceed 5% to 10% total zinc. On mixed gold and silver ores and particularly where the silver is relatively high, the precipitate may run as high as 95% combined gold and silver with only 3% to 5% total zinc.

Owing to the high grade precipitates now generally obtained, most plants in the Western Hemisphere melt direct without preliminary treatment other than partial drying. In Africa, and particularly on the Rand, the "gold slimes" are acid treated and calcined, while in Australia melting is generally preceded by calcining. The purpose o d treatment with sulphuric acid is to remove excess zinc, while calcining, with or without an oxidizing agent such as nitre, is to oxidize the base metals so that they may be slagged off in melting. The above statements apply primarily to gold ores; high grade silver precipitate is invariably melted direct.

TYPES OF MELTING FURNACES

Three types of melting furnaces are in general use, all oil fired. For smaller plants, treating gold ores, the tilting furnace with removable graphite crucible is usually preferred. Clay liners are quite generally used to prolong the life of the pot.

In larger plants, particularly those treating silver ores, tilting furnaces of the reverberatory type are most satisfactory. Such furnaces are provided with a molded hearth of suitable refractory and may be either single or double,

depending on the amount of precipitate to be handled.

In the largest mills, treating either gold or silver ores, stationary reverberatory furnaces are used. These may be built to operate as a conventional reverberatory furnace, with the usual fusion hearth of firebrick, or other suitable refractory, or may be used as reverberatory pot fusion furnaces, with lined graphite pots. This is the furnace generally used in the large mills on the Rand.

Precipitate containing up to 30% to 40% moisture may be fluxed and melted without drying, or dryers, either steam or electric may be used to reduce the moisture to from 15% to 20% before adding flux and charging to the furnace. Where filter presses are used, blowing with compressed air yields a suitable product for melting without further drying.

FLUXES

Fluxes used in melting cyanide precipitate vary somewhat in different parts of the world; the following mixtures are typical but in starting a new plant trial fusions should be made in each case, to determine the most suitable mixture.

For melting raw precipitate from clean gold solutions the following charge will usually give rapid fusions and clean, fluid slags.

75 lb. precipitate	*45 lb. manganese*
(15% to 20% moisture)	*dioxide*
50 lb. borax	*25 lb. silica sand*

Note—Soda bi-carbonate or fluorspar may be substituted for part of the borax, and the manganese may be replaced by nitre.

Silver precipitate, containing 80% or better of silver, is readily melted with the following charge:

100 lb. precipitate	*3 to 5 lb. soda*
(15% to 20% moisture)	*bi-carbonate*
5 to 10 lb. borax	*3 to 5 lb. silica sand*

For melting calcined gold precipitates the following charge is used in many plants:

100 lb. dry calcine	10 lb. soda ash
25 to 40 lb. silica sand	5 lb. fluorspar
40 to 60 lb. borax	

Note—5 to 15 lb. manganese dioxide may be added in some cases.

TESTING SOLUTIONS

The following methods will be found useful for the rapid estimation of gold or silver in barren or precipitated solution. With practice, both estimations can be made with considerable accuracy but are, of course, intended to be entirely supplementary to the regular shift and daily assays.

DOWSETT'S ESTIMATION OF GOLD IN CYANIDE SOLUTION

Take approximately 1000 cc. of solution in a tight stopper jar. (A quart Mason fruit jar is convenient.)

Add saturated solution of NaCy to bring the solution to be tested up to about .10% NaCy.

Add two drops of lead acetate. (Clear saturated solution.)

Add a pinch of zinc dust (approx. 2 grams).

Shake well for about two minutes.

Pour into a large evaporating dish. Settle and decant clear solution.

Add 10 cc. aqua regia

and evaporate nearly to dryness.

Take up in 2 cc. conc. hydrochloric acid.

Pour into small test tube and cool thoroughly.

The tube should be about 3 to 4 inches long and not over ⅛ inch diameter, and the cooling is important.

Incline the tube and add a few drops of fresh, saturated, stannous chloride solution.

The presence of gold will be indicated by a purplish ring at point of contact or by a purplish tinge throughout, if the tube is shaken.

The evaporation may be performed over a spirit lamp or similar flame and does not take over five minutes. The presence of as little as .02 dwt. gold per ton of solution is very plainly shown.

ESTIMATION OF SILVER IN CYANIDE SOLUTION

A few drops of a 10% solution of sodium sulphide added to 25 or 50 cc. of the solution to be tested, gives a pure white precipitate of zinc sulphide in the absence of silver. The precipitate becomes brownish in the presence of silver, and the depth of color is a very close indication of the amount present.

The presence of the usual small amounts of lead in the solution does not affect the result appreciably.

THE ESTIMATION OF OXYGEN IN CYANIDE SOLUTION

During dissolution of the metals, cyanide solutions should of course contain oxygen; before subsequent precipitation of the metals, all of the free oxygen should be removed. Oxygenation is accomplished by contacting the solution or pulp with air in various types of agitators; removal of the free oxygen is effected by the Crowe Process, in which thin films of solution are exposed to a high vacuum in a suitable receiver.

Ordinary water and most cyanide solutions, except those containing unusual amounts of reducing agents, such as sulphocyanide or sulphides, saturate rapidly with oxygen and all cyanide solutions of which we have any knowledge give off all but traces of free oxygen in passing through the Crowe apparatus. Also solutions which have been completely precipitated, either by zinc dust or by zinc shavings, usually contain no free oxygen.

A systematic study of the oxygen content of solutions at various stages of mill operation has, in several cases within our knowledge, led to material improvement and savings. A deficiency in oxygen may be found in agitators or thickeners and the effluent from the Crowe receiver should be checked to make certain that sub-

stantially all of the free oxygen is removed before pre-
tation. The most suitable methods for determining
oxygen in cyanide solutions are given below.

*WHITE'S METHOD

This method was developed by Mr. H. A. White, Con-
sulting Metallurgist for the Union Corporation at Johan-
nesburg, South Africa, and is based upon the color
imparted to an alkaline solution on the addition of pyro-
gallic acid, the "pyro" used in photographic work.

APPARATUS REQUIRED:

1 doz. 250 cc. glass-stoppered bottles
1 burette

CHEMICALS REQUIRED:

*Sodium Hydrate (NaOH) solution, 2N (80 gm. per
liter).*
*Pyrogallic acid or "pyro" (the crystalline salt is
preferable to the powder).*
Brown dye (Diamond Brown or Caramel).

PREPARATION OF STANDARD COLORS

turate a quantity of ordinary hydrant water with
oxygen by passing air through it for an hour. Then, let
stand for another hour to remove bubbles of occluded
air. Fill one of the 250 cc. bottles with this water, add
about 1/10 gm. of pyro and 1 cc. of 2N. NaOH. The
pyro crystals must immediately sink below the surface.
Then insert glass stopper with a twisting motion to ex-
clude any small air bubbles. After adding the soda solu-
tion, the bottle should be full to within a quarter-inch of
the top, so that the stopper may be inserted at a slight
angle. Shake bottle until all the "pyro" is dissolved.

The water will take on a reddish brown color correspond-
ing to the oxygen content of oxygen saturated water at
the existing temperature and pressure. The percentage

*For a full description of this method see *Journal of
Chem. and Met. Soc. of S. A.* (June, 1918) or *Manual
of Cyanidation*, E. M. Hamilton, McGraw-Hill Book
Co.

oxygen content of water, saturated under any given set
of conditions, may be calculated from the table of
Roscoe and Lunt, Volumetric Analysis, Sutton, or from
the chart of Weinig and Bowen shown on page 22.

This color is then matched with a water solution of dia-
mond dye or caramel. A small amount of chromate of
potash will sometimes assist in obtaining the exact shade.

Let us assume that under the existing conditions the
saturation point of water corresponds to 8 mg. oxygen
per liter. Then if standard bottles are made up containing
1 part of the color corresponding to saturation and 7
parts water, this lighter color will correspond to 1 mg.
oxygen per liter and equal parts of standard color and
water will correspond to 4 mg. of oxygen. In the same
way make up a series of eight bottles, the colors of which
represent oxygen contents of from 1 to 8 mg. oxygen
per liter.

ESTIMATION OF OXYGEN—Continued

Fill one of the 250 cc. bottles with the solution to be
tested. Use a rubber tube reaching to the bottom of the
bottle and avoid all agitation. A drop or two of kerosene
oil in the bottle will film the solution and still further
prevent absorption of oxygen. Fill the bottle nearly full,
then add 1/10 gm. pyro and 1 cc. of 2N.NaOH, and
stopper instantly, taking care that no air bubble is left
under the stopper. Shake well and compare with standard
colors.

Some solutions, particularly those resulting from the
cyanidation of silver ores, show fading colors and be-
come cloudy on the addition of pyro and soda.* In such
cases a better comparison is made after standing a def-
inite time, say three to six minutes. Also on such solutions
a better standard color is obtained by making up with
regular plant solution to which is added the usual amount

Oxygen in Cyanide Solutions, E. M. Hamilton, Eng.
& Mining Jour., *July 17, 1920.*

of soda and pyro. Then after standing from three to six minutes, this color is matched with the dye or caramel, and the fractional standards prepared as usual. A small amount of solid pigment, such as yellow ochre, added to the dye or caramel solution will match precipitate which sometimes forms. With such solutions it is better to make up a fresh set of standards whenever oxygen tests are to be made.

This method depends upon the reducing action of a standard sodium hydrosulphite solution on indigo blue. After all the oxygen has been titrated, any excess of hydrosulphite changes the indigo blue to indigo white. The method is said to be accurate to tenths of a milligram per liter of solution, although such accuracy is not required for the purposes under discussion. We refer to the author's paper for a full discussion of the method.

Chart A, Fig. I, is used to determine barometric pressure at various altitudes; Chart B is used to determine standard saturation values for various temperatures and pressures. In Chart A, altitudes, in feet, are plotted on the horizontal axis and pressures, in millimeters, on the vertical axis. To find the pressure corresponding to a certain altitude, follow the elevation line downward to its intersection with the curve X, then horizontally to the right and read the pressure. For example, if the elevation is 6000 ft., the 6000-ft. line is followed to its intersection O with the curve X, then the corresponding pressure, 607 mm., is obtained from the right-hand side of the chart.

**Determination of Dissolved Oxygen in Cyanide Solutions.* A. J. Weinig and Max W. Bowen. Trans. Am. Inst. Mining & Met. Engs., *Sept., 1924.*

In Chart B, temperatures are plotted on the horizontal axis and the amount of oxygen, in milligrams per liter of distilled water, is plotted on the vertical axis; various pressure curves also are plotted, as shown. To find the saturation value for a certain temperature and pressure follow the temperature line upwards until the point corresponding to a given pressure is reached, then follow horizontally across to the left-hand side of the chart and read off the amount, in milligrams, of oxygen per liter of solution. For example, to determine the amount of oxygen in a solution having a temperature of 59° F., at an elevation of 6000 ft., it has already been found that at an elevation of 6000 ft. the pressure is 607 mm.; therefore the 607-mm. pressure curve must be used; that is, it is necessary to interpolate between the 600-mm. and the 650-mm. curves. The 59° F. line is followed to its intersection R with the 607-mm. curve, then from the left-hand side of the chart is read off 8 mg. of oxygen per liter. The same procedure is used for the various pressures, using the curve corresponding to the particular pressure. For any particular plant, a solubility curve for that elevation should be plotted.
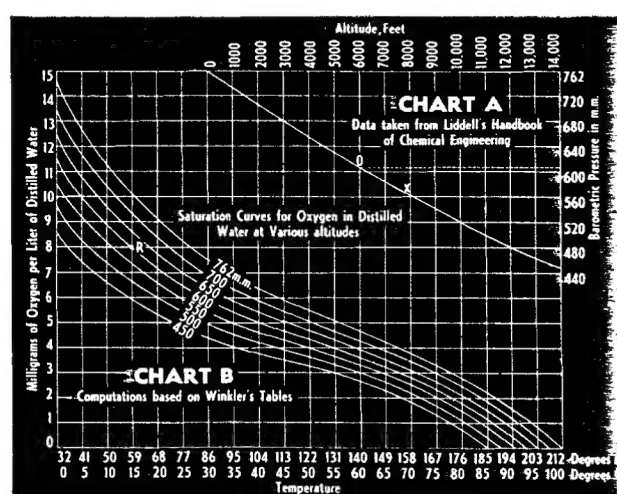

FIGURE I

CROWE'S LABORATORY PRECIPITATION METHOD:

The following method of precipitating cyanide solutions will frequently be found useful in the testing laboratory. It depends upon the elimination of dissolved oxygen from the solution by the addition of a chemical reducing agent which may be either sodium or calcium hydrosulphite. While the reactions involved are not strictly comparable with actual plant practice, the results will be found to check closely with those obtained by mechanical de-aeration of the solution as practiced in the Merrill-Crowe Process.

Complete precipitation of gold and silver from cyanide solutions with zinc dust requires that all free oxygen be first removed or neutralized. The precipitant itself can accomplish this but with a wasteful consumption of zinc. Exposure to a high vacuum removes from 90% to 95% the oxygen and the remainder is consumed by reaction with the zinc or by the addition of a small amount of hydrosulphite. As a laboratory procedure it is simpler to add sufficient of the reducing agent to combine with all of the dissolved oxygen initially present or which may be introduced during the test.

METHOD A

To 1000 cc. of the solution to be precipitated add 150 mg. of sodium hydrosulphite. Then emulsify 100 mg. of zinc dust in 10 cc. of distilled water to which has been added 10 mg. of lead nitrate.
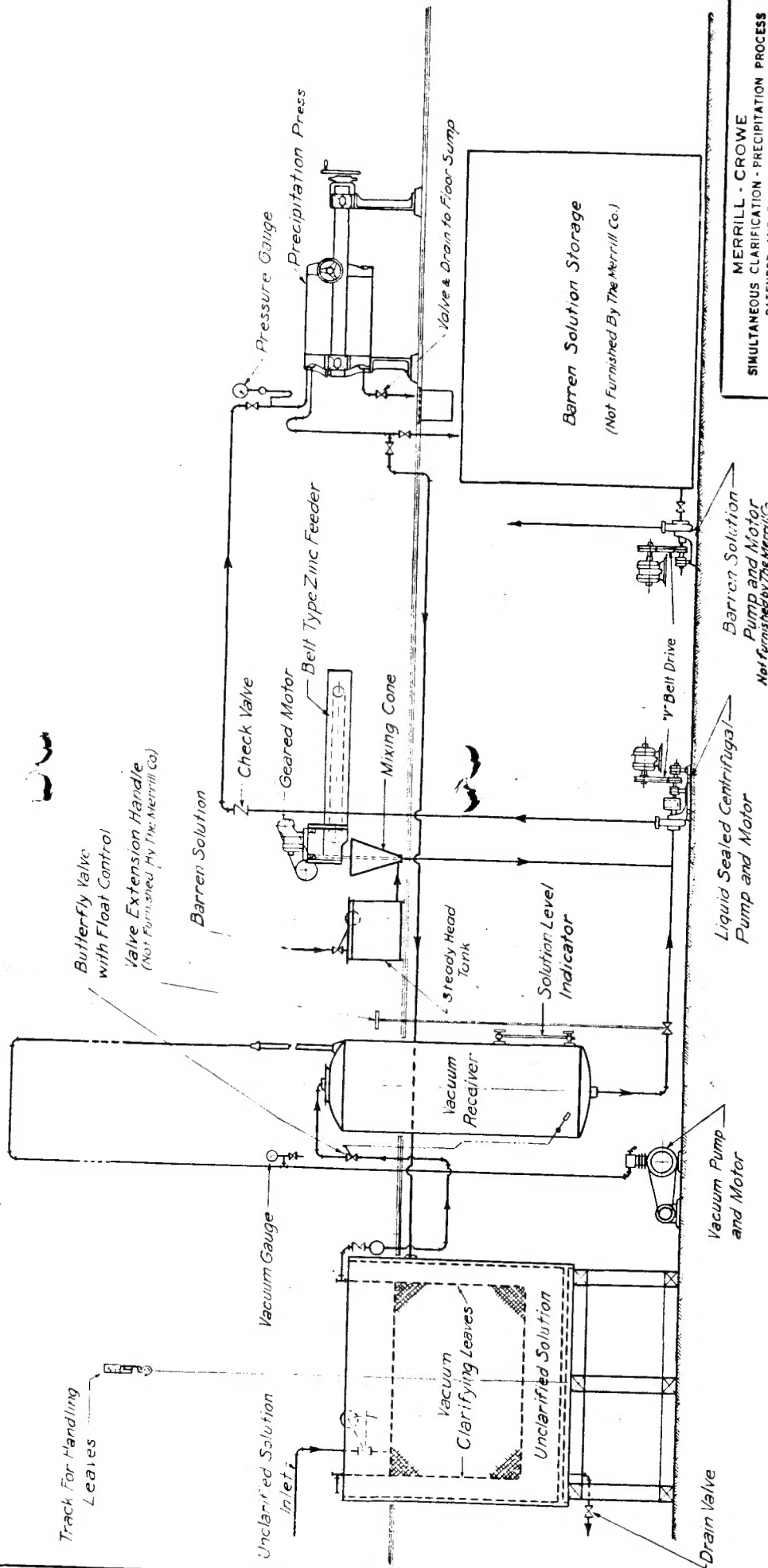
Add the emulsion to the solution and stir intermittently for 30 minutes. Filter rapidly on a 2-inch Buchner filter with double filter paper which has been suitably pre-coated with paper pulp or diatomaceous silica.

N.B. *The solution should be stirred gently to minimize introduction of air, and the filtration stopped before all the solution is through, this also to prevent aeration.*

If the solution contains appreciable amounts of silver or copper, the zinc dust should be increased accordingly.

METHOD B

Dissolve 100 mg. of zinc dust in 20 cc. of distilled water, saturated with sulphur dioxide. Neutralize with a slight excess of calcium hydrate and filter on a small filter. Add the clear filtrate of calcium hydrosulphite to 1000 cc. of the solution to be precipitated. Then add the lead-zinc emulsion and proceed as in Method A.

Track For Handling Leaves

Unclarified Solution Inlet

Vacuum Gauge

Butterfly Valve with Float Control

Valve Extension Handle
(Not Furnished By The Merrill Co.)

Barren Solution

Pressure Gauge

Precipitation Press

Valve & Drain to Floor Sump

Check Valve

Geared Motor

Belt Type Zinc Feeder

Mixing Cone

Barren Solution Storage
(Not Furnished By The Merrill Co.)

Steady Head Tank

Solution Level Indicator

Liquid Sealed Centrifugal Pump and Motor

"V" Belt Drive

Barren Solution Pump and Motor
Not Furnished by The Merrill Co.

Vacuum Receiver

Vacuum Clarifying Leaves

Unclarified Solution

Vacuum Pump and Motor

Drain Valve

NOTE: Interconnecting Piping Not Furnished by The Merrill Co.